



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

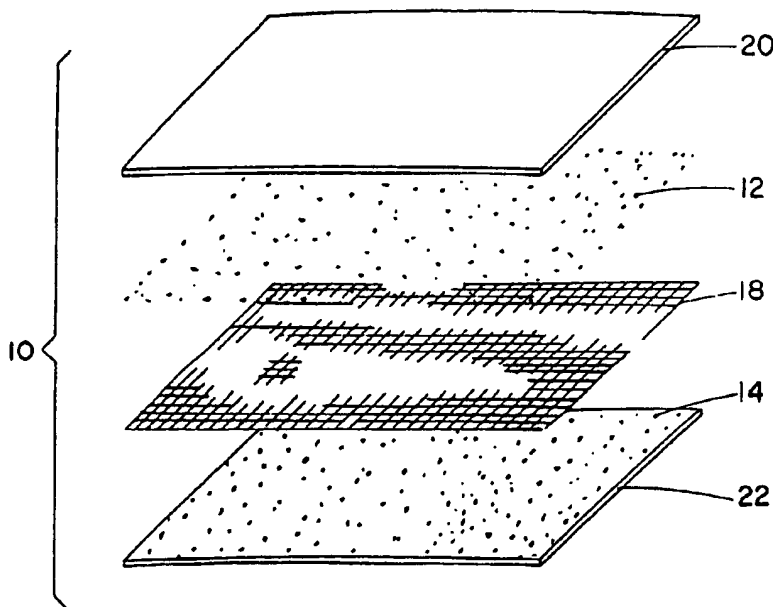
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(21) International Application Number: PCT/US94/05570 (22) International Filing Date: 18 May 1994 (18.05.94) (30) Priority Data: 08/064,391 18 May 1993 (18.05.93) US (71) Applicant: STERIS CORPORATION [US/US]; 9450 Pincen- dle Drive, Mentor, OH 44060 (US). (72) Inventors: MALCHESKY, Paul, S.; 239 Barrington Ridge, Painesville Twp, OH 44077 (US). KRALOVIC, Raymond, C.; 3703 Edgewater Drive, Ashtabula, OH 44004 (US). (74) Agent: KOCOVSKY, Thomas, E., Jr.; Fay, Sharpe, Beall, Fagan, Minnich & McKee, 1100 Superior Avenue, Suite 700, Cleveland, OH 44114-2518 (US).			(81) Designated States: AU, CA, JP, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  Published With international search report. Before the expiration of the time limits for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: CONTROLLED OXYGEN/ANTI-MICROBIAL RELEASE FILMS

## (57) Abstract

A film for releasing at least one of an antimicrobial agent, oxygen, and a medicament includes a flexible, porous layer (18) such as a woven, non-woven, or knitted cloth or a layer of open cell foam. A first dry reagent (12) and a second dry reagent (14) which react in the presence of a dilutant to form the anti-microbial agent, oxygen, or medicament attached to the flexible, porous layer. In one preferred embodiment, the two dry reagents are disposed on opposite sides of the flexible, porous layer such that the flexible porous layer keeps the two apart and prevents a premature reaction. Porous outer layers (20, 22) prevent the powdered reagents from being wiped off while permitting dilutant access. In a preferred embodiment, the powdered reagents include acetylsalicylic acid and a perborate which react in the presence of water to generate peracetic acid (an antimicrobial agent which breaks down in a matter of minutes to hours into oxygen) and salicylic acid (a topical keratotic).

The rate at which the reaction occurs and the peracetic acid breaks down into oxygen is controlled by buffering the pH of the powdered reagents, by selectively micro-encapsulating the powdered reagents, by controlling the porosity of the layers, or the like. Optionally, surfactants, detergents, emollients, gels, and the like can be added to the dry reagents. Alternately, a single reagent which releases oxygen or forms a strong oxidant may be used.



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**CONTROLLED OXYGEN/ANTI-MICROBIAL RELEASE FILMS****Background of the Invention**

The present invention relates to the microbial decontamination arts. It finds particular application with bandages and wipes and will be described with particular reference thereto. It is to be appreciated, however, that the present invention will also find application in other areas where oxidants, anti-microbial agents, or medicaments are generated *in situ* such as in gloves, drapes, and the like.

Heretofore, various wipes and other cloth-like materials have been impregnated with an anti-microbial agent. The anti-microbial agent, most often in a liquid form, is coated on, caused to soak into, or otherwise attached to a flexible carrier or film. Often, the anti-microbial agent treated film is packaged in a sealed pouch to prevent evaporation or contamination. The pouch is opened to use the wipe. The wipe is typically packaged with a sufficient amount of a liquid carrier that a wet layer of carrier and anti-microbial agent are left on the wiped surface. Alternately, the carrier or a dilutant such as water may be added to the film when the package is opened to render the anti-microbial agent more mobile.

Flexible fabric or fiber-like materials which are apt to support mildew, mold, or bacterial growth are often treated with an anti-microbial agent. Often, the fabric or fiber carries materials, e.g., sizing which are apt to support such microbial growth. The anti-microbial agent is

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typically intermixed with the material which is apt to support microbial growth.

One of the problems with wipes and films of this type is that the anti-microbial agent requires a long shelf life. The wipe may be in the package for weeks or months before it is opened. After the wipe is used and discarded, a significant amount of the anti-microbial agent remains in the wipe. If the anti-microbial agent is a toxin or poison with a long life, disposal of the used wipe carries undesirable environmental and ecological side effects.

The present invention contemplates a new and improved material which overcomes the above-referenced problems and others.

#### Summary of the Invention

In accordance with one aspect of the present invention, a film carries one or more constituents which in the presence of a dilutant form oxygen or a strong oxidant.

In accordance with a more limited aspect of the present invention, the film is one of a woven, non-woven, or knitted fibers, a flexible foam material, or combinations thereof.

In accordance with another aspect of the present invention, the film carries at least two reagents that react in the dilutant to form the oxygen or anti-microbial agent. A means is provided to maintain the constituents or reagents separated until contacted with a dilutant.

In accordance with one more limited aspect of the present invention, the means for keeping the two constituents separated includes one of a filter, micro-encapsulation, and immobile implantation of the reagents into displaced first and second layers.

In accordance with another aspect of the present invention, means are provided for controlling a reaction rate between the constituents to control the rate at which oxygen or anti-microbial agent is produced.

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In accordance with a more limited aspect of the present invention, the means for controlling the reaction rate includes added buffers which increase the pH to accelerate the reaction rate or decrease the pH to retard the reaction rate.

In accordance with another aspect of the present invention, the first reagent includes an acetyl donor and the second reagent includes a perborate. Suitable acetyl donors include acetylsalicylic acid which react with a perborate, preferably sodium perborate monohydrate or sodium perborate anhydrous to form peracetic acid and salicylic acid. The peracetic acid is a strong oxidant which decomposes to liberate free oxygen. The salicylic acid is a keratotic. Other suitable acetyl donors include tetraacetyl ethylenediamine (TAED), diacetyl dioxohexahydratriazine (DADHT), tetraacetyl glycoluril, and sodium nonanoyl oxygenzene sulfonate.

In one method of use, the film material is formulated such that the constituents react quickly, i.e. have a very short half-life. The film or the surface to be disinfected are wet, such as by spraying or dipping. The dilutant allows the constituents to react, generating the anti-microbial agent or oxidant. Preferably, the half-life of the anti-microbial agent or oxidant is only a minute or two.

In accordance with another aspect of the present invention, the film is incorporated in a bandage and configured such that the reaction progresses very slowly, i.e. there is a long half-life. The bandage is placed over a wound. Moisture from the wound acts as the dilutant allowing the reaction which produces the anti-microbial agent and/or oxidant to take place. Preferably, the half-life is on the order of several hours to a day or two such that the anti-microbial agent or oxygen is produced substantially continuously over the time that the dressing is applied to the wound.

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One advantage of the present invention is that it has a relatively long shelf-life and a relatively short half-life.

Another advantage of the present invention is that the anti-microbial agent or oxidant is generated *in situ* at a controllable rate.

Another advantage of the present invention is that it effectively kills microbes yet is not polluting when discarded.

Still further advantages of the present invention will become apparent to those of ordinary skill in the art upon reading and understanding the following detailed description of the preferred embodiments.

#### Brief Description of the Drawings

The invention may take form in various components and arrangements of components, and in various steps and arrangements of steps. The drawings are only for purposes of illustrating a preferred embodiment and are not to be construed as limiting the invention.

FIGURE 1 illustrates a film impregnated with dry reagents which react in the presence of a dilutant to form an anti-microbial agent or oxidant;

FIGURE 2 illustrates another embodiment of the present invention in which reagent particles are separated by a filter;

FIGURE 3 illustrates another embodiment of the present invention incorporated into a wipe;

FIGURE 4 illustrates a wound dressing in accordance with the present invention;

FIGURE 5 illustrates another embodiment of the present invention which may advantageously be incorporated into the dressing of FIGURE 4;

FIGURE 6 illustrates yet another embodiment of the present invention in which the reagents are micro-encapsulated;

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FIGURE 7 illustrates a glove incorporating the present invention; and,

FIGURES 8 and 8A illustrate a brush incorporating the present invention.

5        Detailed Description of the Preferred Embodiments

With reference to FIGURE 1, a film 10 is impregnated with a first reagent or constituent 12 and a second reagent or constituent 14, which reagents react in situ in the presence of a dilutant to form an anti-  
10        microbial agent, a strong oxidant, or oxygen. In one preferred embodiment, one of the reagents 12 includes a sodium or other perborate in dry form or other dry constituents which liberate oxygen. Sodium perborate monohydrate and sodium perborate anhydrous are preferred.  
15        The other reagent 14 includes a dry acid precursor. The dry acid precursor and the dry persalt react when dissolved in water or other appropriate dilutants to form a strong oxidant. Further to the preferred embodiment, the acid precursor is an acetyl donor, such as acetylsalicylic acid,  
20        tetraacetyl ethylenediane (TAED), diacetyl dioxohexahydratriazine (DADHT), tetraacetyl glycoluril, and sodium nanonoyl oxygenzene sulfonate. Acetylsalicylic acid and sodium perborate react in water to form peracetic acid, salicylic acid, and sodium metaborate. Peracetic acid is  
25        a strong oxidant with a relatively short half-life that liberates free oxygen as it breaks down. Salicylic acid is a topical keratotic which softens or dissolves horny layers of the epidermis such as warts, callouses, and dead skin. In addition to softening skin, salicylic acid has anti-  
30        microbial properties.

Single constituent systems which react in a solvent, such as water, to release oxygen are also contemplated. Preferred single constituents include stable solid acetyl peroxyborate, magnesium monoperoxy phthalate,  
35        and urea peroxide.

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In use, the film 10, an open cell foam in the embodiment of FIGURE 1, is brought into contact with water or other dilutant such that the physically displaced implanted dry acid precursor and dry persalt or the single constituent are dissolved and react forming an antimicrobial agent and liberating oxygen. When used as a wipe, the film may be dipped or sprayed with water to activate it. Alternately, the surface to be disinfected may be wet such that the film and the impregnated reagents become wet, dissolve, and react from the water on the surface. When used as a bandage, the film may be initially dampened, such as with spray, to start the reaction. The reaction may be continued or in some embodiments initiated by moisture from the wound. When used as a dressing, the oxidant disinfectant agent not only functions as an antimicrobial agent to prevent infection, but also liberates free oxygen which promotes healing.

With reference to FIGURE 2, rather than implant the dry constituents or reagents 12, 14 physically displaced from each other, other means can be provided for maintaining the dry constituents separated until activated by the dilutant. In the embodiment of FIGURE 2, particles of the first reagent 12 and particles of the second reagent 14 are separated by a filter sheet 18. The filter sheet has a pore size which is sufficiently small relative to the particle size of the two powdered reagents that the particles are maintained physically separated. Yet, once dissolved in the dilutant, the pore size is sufficiently large that the dilutant and dissolved materials pass therethrough and react. A top or first outside coating layer 20 and a bottom or second outside coating layer 22 shield the particles of the two reagents from physical interaction so that they are not lost or brushed off before the film is actuated with the dilutant. At least one of the two covering layers is permeable to the dilutant to allow ready access to the constituents.



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It is also advantageous to control the reaction rate such that the rate at which the anti-microbial agent or oxygen is produced occurs substantially at a preselected rate. In the embodiment of FIGURE 2, a means for controlling the reaction includes the pore size of the filter material 18. In one embodiment, the reaction rate is controlled by limiting communication between the constituents. The means for controlling the reaction rate further includes the permeability of the covering layers 20 and 22. By limiting the rate at which the dilutant can reach the dry constituents, the rate at which there is sufficient dilutant to allow them to react is controllable. In another embodiment, the means for controlling the reaction rate includes the addition of further powdered reagents. In particular, the reaction between the preferred acetylsalicylic acid and sodium perborate is pH sensitive. At a high pH, the reaction occurs quickly. When the pH is buffered such that it remains near neutral even when the peracetic acid is produced, the reaction proceeds more slowly. The peracetic acid buffered nearer neutral remains stable for a relatively long duration, generally on the order of hours, rather than breaking down into oxygen quickly as it does when the pH is high. Lower pH serves as a stabilizer. Analogously, temperature affects the reaction. The maximum yields of peracetic acid are higher at 20° C while the times needed to reach the maximum are longer at lower temperature. The time for converting TAED to peracetic acid is longer than for DADHT.

With reference to FIGURE 3, the film 10 includes readily porous upper and lower layers 20, 22 between which the powdered reagents are contained. A dry, moisture absorbing gel material 24, such as sodium silicate, separates the dry first reagent 12 and second reagent 14. The dry gel 24 absorbs moisture strongly forming a wet slush which permits the powdered constituents 12 and 14 to mix and react. A hermetic seal, such as a plastic or cellophane package or a foiled pouch 26 encompasses and

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seals the film 10 to prevent moisture from being absorbed by the gel and starting the reaction prematurely. The gel may be formulated chemically or configured physically, such as by the addition of a porous fabric layer, to control the rate at which the dry reagents are dissolved and intermix to react. Further, the dry reagents may again be encapsulated to control the reaction rate. pH buffers or other reaction controlling constituents are preferably physically intermixed with either the first or the second powdered reagent or the gel. The dry reagents of the wipe may also include surfactants or wetting agents, detergents, moisture absorbing gels to remove water from the wiped surface, and the like.

With reference to FIGURE 4, a wound dressing 30 includes a section of the film 10 disposed between a gauze or other porous wound contacting layer 32 and a covering layer 34. Preferably, the covering layer 34 includes an adhesive layer 36 which adheres to the film 10 to a central portion thereof, adheres the wound contacting portion 32, and adheres to the patient's skin around the wound. The film 10 may have the construction set forth in any of the preceding FIGURES or in those described below. In one preferred embodiment, the film 10 has substantially the construction of FIGURE 2. The lower layer 22 disposed towards the patient's skin is porous to moisture exiting the wound. Lower layers of different porosity are used for different types of wounds. For example, the bandage is advantageously used to generate oxygen to promote the healing of a wound. The lower layer 22 has a porosity which permits water vapor to pass therethrough at a controlled rate. The water vapor causes a limited wetting of the dry reagents such that they dissolve gradually and react, at an analogous controlled rate. Preferably, the powdered constituents are buffered to have a relatively high pH such that the preferred acetylsalicylic acid and perborate form a peracetic acid with such a high pH that it is relatively unstable and breaks down quickly to free

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oxygen. A pH of 11 or higher is preferred for a quick breakdown to oxygen. The porosity of the lower layer 22 is again permeable to the free oxygen such that it enters the wound. The upper layer 20, being redundant with the layer 34 may be eliminated. The layer 34, and layer 20 if redundantly applied, may be moisture and gas permeable or may be semipermeable to either moisture or gas. Venting allows excess water vapor to escape. In some applications, layer 34 is impermeable.

For other types of wounds, the generation of a strong anti-microbial agent is important as well as the production of oxygen. The porosity of lower layer 22 is again selected to control the amount of moisture permitted to enter. If the dressing is to be applied for a relatively short duration, e.g. a few hours, the lower layer 22 is preferably permeable by liquids such as water vapor from the skin, liquids exiting the wounds, or water sprayed from a spray bottle to actuate the dressing.

The preferred acetylsalicylic acid and perborate react to form not only peracetic acid, but also salicylic acid. Preferably, the lower layer 22 is sufficiently porous that it allows the dilutant with dissolved salicylic acid to flow back into the wound to promote healing. As another alternative, the salicylic acid can be used to remove horny layers of the epidermis. The film may be wet, such as by spraying or dipping and then applied to the area to be treated. Preferably, the dry constituents include a gel which holds the water and continues the reaction and permits the salicylic acid solution to continue to reach the horny layer for several hours to a day. For relatively longer stability, an after reaction pH of 9.2-10 is preferred. The dry reagents 12 and 14 may also include emollients and other skin softeners.

With reference to FIGURE 5, the constituents 12 and 14 are carried by layers of woven, non-woven, or knitted fibers or open cell or foam 40, 42. The dry constituents are sprayed and dried, impregnated, dry

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sprayed, or otherwise attached to or incorporated into the fibers or foam layers. A filter layer 18 holds the dry particulates apart. Outer layers 20 and 22 contain and protect layers 40 and 42. At least one of the outer layers is fluid permeable such that the dilutant can penetrate, dissolve the dry constituents, and start the chemical reaction. The porosity of the filter 18 and the outer layers 20, 22 control the rate at which the dilutant can enter and the reagents intermix, hence the rate of reaction. Additional buffering compositions may also control the rate of reaction. Surfactants, detergents, emollients, and the like may also be included in dry form.

In the embodiment of FIGURE 6, the single constituent or one or both of the dry reagents 12 and 14 are micro-encapsulated individually. The thickness and the type of the micro-encapsulation controls the reaction rate. For a longer reaction rate, some of the micro-encapsulations are thin to react quickly, others thicker to react more slowly, and others thicker yet such that an extended duration is required before the dilutant penetrates the encapsulation and reaches the dry constituent. The micro-encapsulated constituents or reagents are between outer layers 20, 22. At least one of the outer layers is porous to permit the dilutant to enter and the resultant anti-microbial agent, oxidant, or free oxygen to exit.

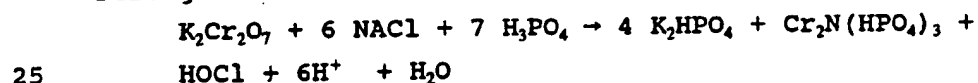
With reference to FIGURE 7, the constructions of the preceding embodiments can be fabricated into various articles. For example, a protective glove 50 is defined by a continuous impermeable hand-shaped layer 52 of rubber, plastic, or other film material which is impermeable to the dilutant, the anti-microbial agent, strong oxidants, and gaseous oxygen, as well as any emollients, detergents, or other substances which may be dissolved in the dilutant. The film 10 is adhered to the outer surface of all or selected portions of the glove. The film 10 is preferably laminated over the palm, thumb, and finger pad portions of

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the glove, i.e. the portions of the glove which would contact a grasped or touched object. An outer, permeable layer 22 surrounds the film 10 protecting it. Preferably, the outer layer 22 is a material which can be readily  
 5 affixed to the impermeable liner 52, such as by heat fusion, adhesives, or the like. Preferably, the film 10 has periodic discontinuities where the inner and outer layers contact and adhere to each other.

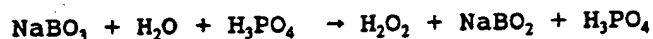
With reference to FIGURES 8 and 8A, the single or  
 10 the plural constituents 12, 14 are impregnated in bristles 60 of a brush 62. The bristles may be natural or synthetic fibers or other constructions which securely hold the dry constituents. When the brush is dipped into a dilutant, the constituents react forming an anti-microbial for  
 15 scrubbing a surface.

Although the preferred embodiment uses an acetylsalicylic acid and sodium perborate reaction, other oxidizing or antimicrobial agents can also be generated *in situ*, such as chlorine dioxide, chlorine, hydrogen  
 20 peroxide, and mixtures thereof. More specifically, potassium chromates, sodium chloride, and phosphates may be mixed according to the following equation to produce a strong chlorine oxidant on the addition of water:

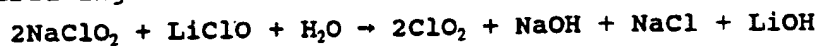


Optionally, excess dichromate and an organic corrosion inhibitor may be provided for improved buffering and corrosion inhibiting.

Hydrogen peroxide and an inorganic inhibitor can  
 30 be generated:



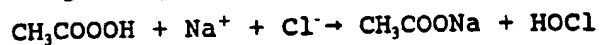
Similarly, chlorine dioxide can be generated from powdered ingredients on the addition of water:



35 A mixed biocide system can be achieved by adding sodium chloride to the peracetic acid reaction to produce hypochlorous acid. Because sodium chloride is a component

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of physiological fluids, the reaction can be partially physiologically regulated.



Excess peracetic acid is deliberately present such that  
5 both peracetic acid and hypochlorous acid are present in the biocidal solution.

Other reagents include perborates which react in water to liberate free oxygen, and constituents which react to form other medically useful compositions.

10 The invention has been described with reference to the preferred embodiment. Obviously, modifications and alterations will occur to others upon reading and understanding the preceding detailed description. It is intended that the invention be construed as including all  
15 such modifications and alterations insofar as they come within the scope of the appended claims or the equivalents thereof.

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Having thus described the preferred embodiment, the invention is now claimed to be:

1. A film including a porous layer further characterized by:

at least one dry constituent held in the porous layer, which dry constituent reacts in a dilutant to generate at least one of oxygen and a strong oxidant.

2. The film as set forth in claim 1 further characterized by the at least one dry constituent including:

a first dry reagent substantially constrained in association with the porous layer; and

a second reagent constrained in association with the porous layer, the first and second reagents reacting in the presence of the dilutant *in situ* to create a solution containing the at least one of oxygen and a strong oxidant.

3. The film as set forth in claim 2 further characterized by a means for maintaining the first and second dry reagents separated in the absence of the dilutant.

4. The film as set forth in claim 3 further characterized by the means for maintaining the first and second reagents separated including at least one of:

separately encapsulated particles of the first and second dry reagent;

a filter material having pores smaller than a physical size of particles of first and second dry reagents physically separating the particles of the dry reagents;

the first dry reagent being adhered to a first flexible layer and the second dry component being adhered to a second flexible layer, the first and second flexible layers being disposed on opposite sides of the porous layer; and

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15       impregnating particles of the first and second  
dry reagents displaced from each other in a common flexible  
layer.

5       5. The film as set forth in any one of  
preceding claims 2, 3, and 4 further characterized by the  
first reagent including an acid precursor and the second  
reagent including a perborate, the acid precursor and  
perborate reacting in the presence of water to generate the  
strong oxidant and further including at least one of:

10           a surfactant;  
            a detergent;  
            an emollient;  
            a pH buffer; and  
            a water absorbing gel.

6. The film as set forth in any of claims 2, 3,  
4, and 5 further characterized by the first reagent  
includes an acetyl donor and the second reagent includes a  
perborate such that peracetic acid is generated.

5       7. The film as set forth in claim 6 further  
characterized by the acetyl donor including at least one of  
acetylsalicylic acid, tetraacetyl ethylenediamine, diacetyl  
dioxohexahydratriazine, tetraacetyl glycoluril, and sodium  
nanononyl oxygenzene sulfonate.

8. The film as set forth in any of preceding  
claims 2-7 further characterized by a means for controlling  
a reaction rate between the first and second reagents.

5       9. The film as set forth in claim 8 further  
characterized by the reaction rate controlling means  
controlling the reaction rate to cause a complete reaction  
in less than 5 minutes, whereby the strong oxidant is  
generated rapidly enabling the film to be used as an  
anti-microbial wipe.



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10. The film as set forth in any one of the preceding claims further characterized by the porous material including at least one of:

- 5 woven fibers;
- non-woven fibers;
- knitted fibers;
- a permeable synthetic sheet; and
- a flexible foam layer.

11. The film as set forth in any one of the preceding claims further characterized by a patient dressing disposed adjacent one side of the porous material, the patient dressing being adapted for direct patient contact over a wound or region to be treated; and,

5 an adhesive layer for adhering the porous layer and the patient dressing to the patient such that generated oxygen promotes healing.

12. The film as set forth in any one of the preceding claims further characterized by the film being affixed to an exterior of a dilutant impermeable glove.

13. A method of using the film of claim 1 characterized by:

- 5 wetting the film with the dilutant such that the reaction progresses and bringing the film into contact with a surface to be treated.

14. The method as set forth in claim 13 further characterized by:

- 5 adhering the film to a patient and wherein the wetting step includes wetting the film with moisture emitted from the patient such that moisture from the patient causes the reaction.

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15. The method as set forth in claim 13 further characterized by:

the film being wet by at least one of immersion or spraying and the film is wiped over the surface.

16. The method as set forth in claim 15 further characterized by:

the generating of at least one of oxygen and a strong oxidant includes reacting first and second dry  
5 reagents and a dilutant within the film to form the oxygen or strong oxidant solution.

17. The method as set forth in claim 16 further characterized by:

controlling a rate at which the first and second reagents react to control a rate at which the oxygen or  
5 strong oxidant is generated.

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FIG. 1

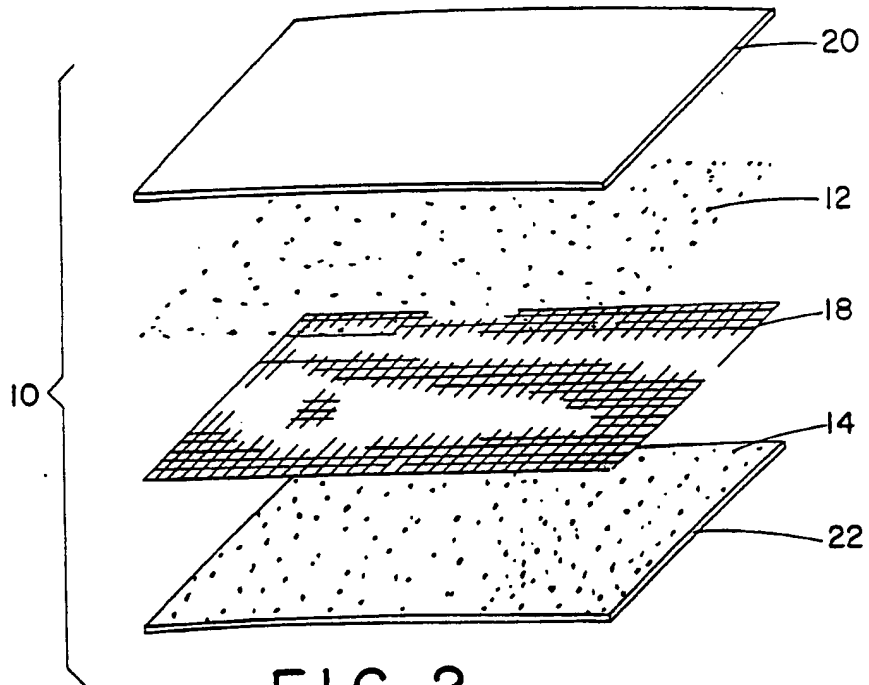
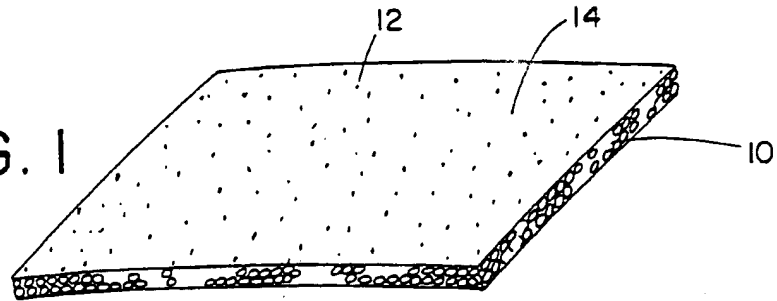


FIG. 2

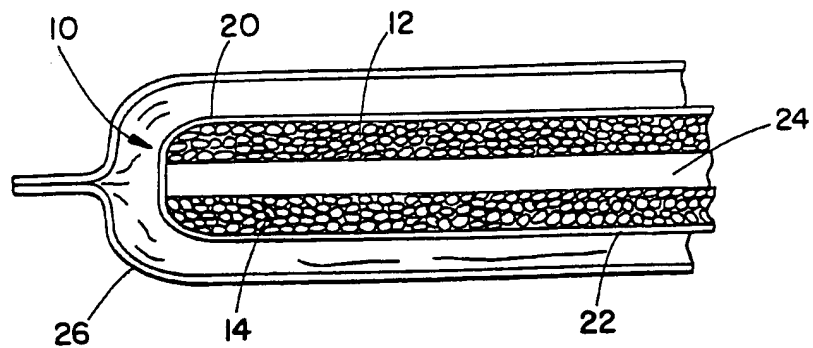


FIG. 3

SUBSTITUTE SHEET (RULE 26)

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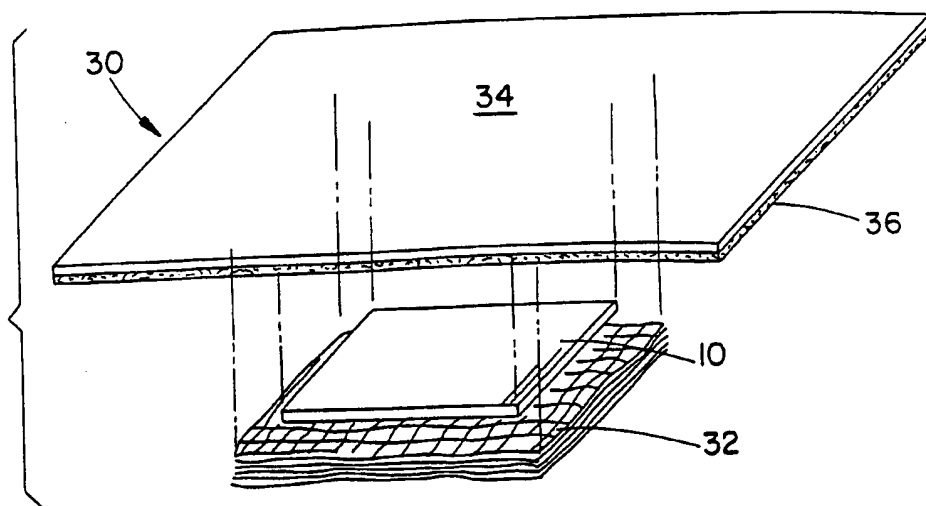


FIG. 4

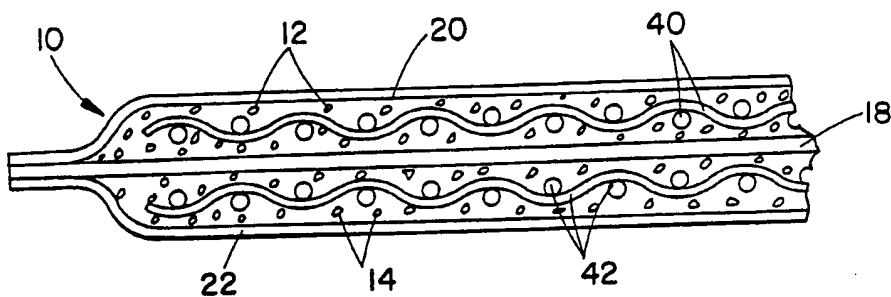


FIG. 5

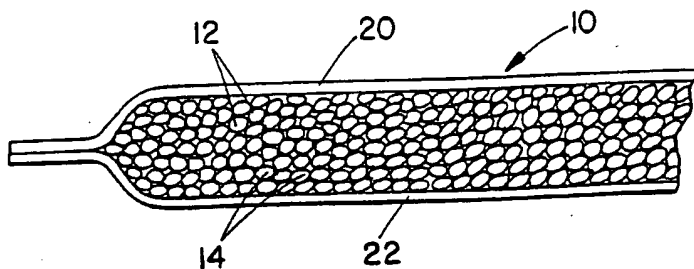


FIG. 6

SUBSTITUTE SHEET (RULE 26)

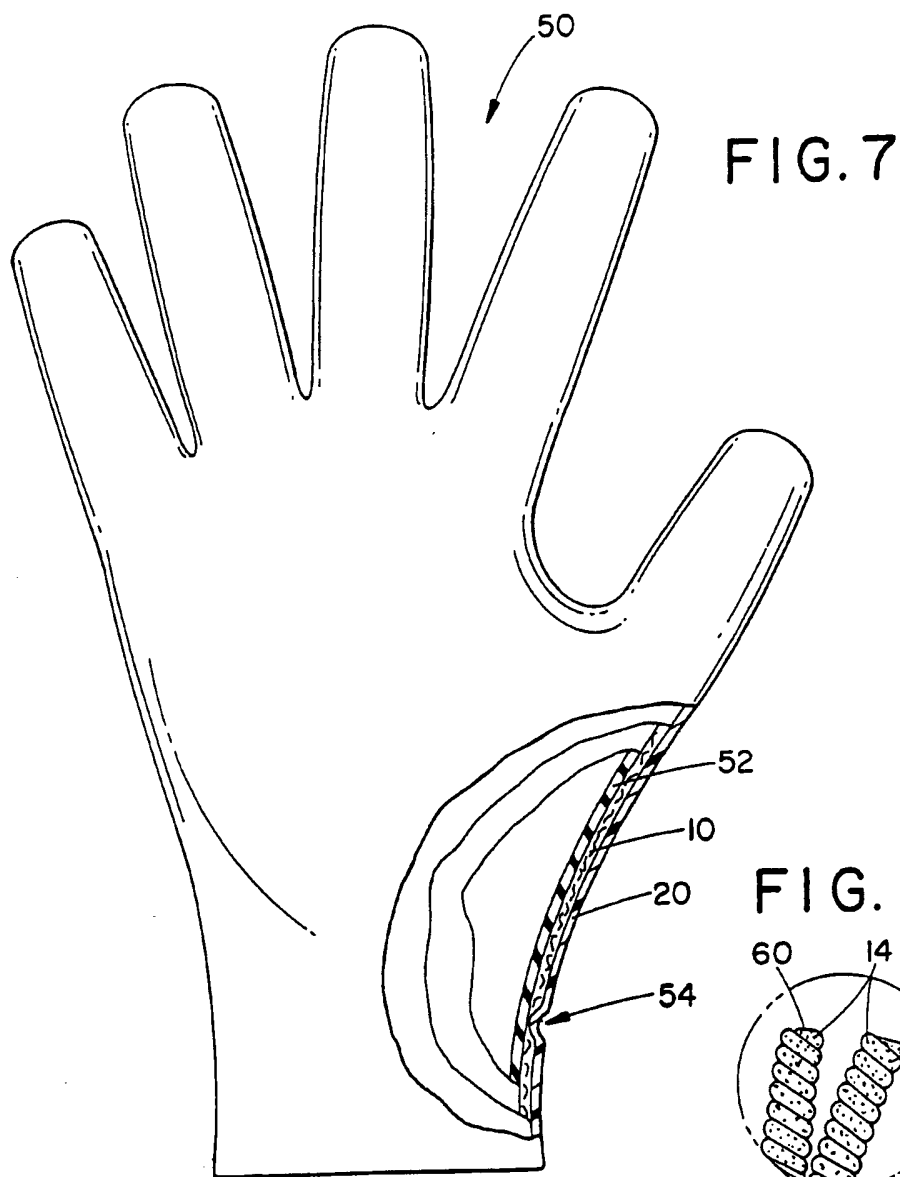


FIG. 8A

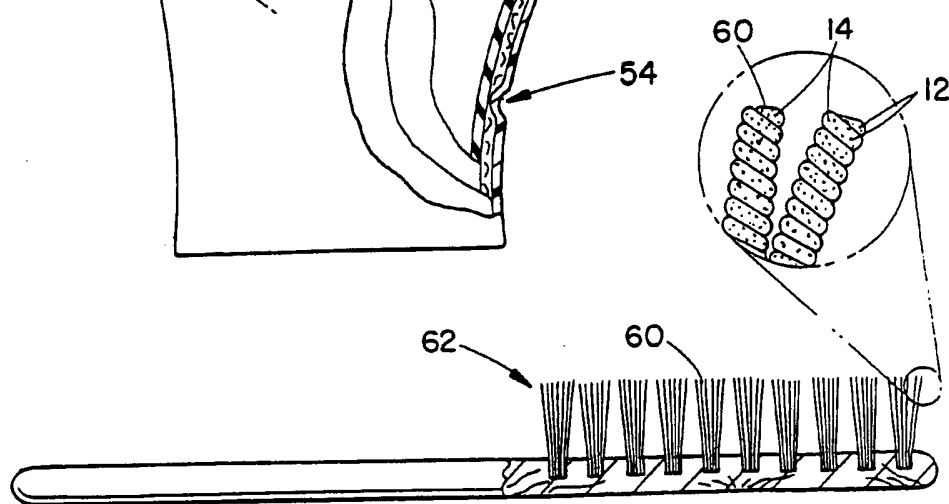


FIG. 8

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US 94/05570

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 5 A61L2/16 A61L15/46 A61L15/44 A61L31/00 A01N25/34  
A61L2/20

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 A61L A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US,A,5 104 660 (BRUCE A. BARBER.) 14 April 1992 see column 2, line 39 - line 68 see column 3, line 1 - line 29; examples ---	1-5,8, 10,11,14
X	US,A,4 847 089 (DAVID N. KRAMER; PHILIP A. SNOW) 11 July 1989 see column 1, line 44 - line 47 see column 4, line 6 - line 34 see column 7, line 7 - line 16 see column 7, line 53 - line 56 see column 8, line 19 - line 26; examples --- -/--	1,5,9, 10,13

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

## \* Special categories of cited documents:

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Date of the actual completion of the international search

21 September 1994

Date of mailing of the international search report

07.10.94

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ESPINOSA, M

## INTERNATIONAL SEARCH REPORT

 International application No.  
 PCT/US 94/05570

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
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X	EP,A,0 351 907 (UNILEVER NV) 24 January 1990 see column 2, line 36 - line 45 see column 3, line 49 - line 58 see column 4, line 32 - line 36; claims; examples ---	1,9,10, 13
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